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INVESTIGATIONS OF CARBIDES AS CATHODES
FOR THERMIONIC SPACE REACTORS

Informal Monthly Report for the Period
March 1, 1963 through March 31, 1963



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Project No. 306
National Aeronautics and Space Administration
Contract No. NAS 3-2532

April 29, 1963

SUMMARY

The work carried out under Contract NAS 3-2532 during March, 1963 is summarized below:

1. Long-term Tests of the Vaporization and Emission of UC-ZrC and W-clad UC in Cesium Vapor.

1.1 Rate of Vaporization of UC-ZrC in Cesium Vapor.

Another 100-hr run has been made at 2083°K in cesium vapor at a pressure of 1.8 torr with a nominal 90 UC - 10 ZrC sample B₁-3 of 95.5% theoretical density*.

1.2 Emission Stability of UC-ZrC and W-clad UC in Cesium Vapor.

Test of the first life-test cell containing a 90 UC - 10 ZrC sample (A₃-1) was discontinued after about 500 hr because leakage developed in the tantalum partition between the cell and the electron-gun compartment, and in the tantalum sheath of the W-(W-Re) thermocouple. The second life-test cell is being assembled. A UC wafer clad with vapor-deposited tungsten will be used as the cathode.

2. Studies of the High-Temperature Properties of UC-ZrC.

2.1 Effect of Porosity on the Vaporization and Electron Emission Properties of UC-ZrC in Vacuum.

Measurements have been completed on the vacuum rate of vaporization of a low-density** 30 UC - 70 ZrC (nominal composition) sample B₁-9 made by cold-pressing and sintering. The vacuum emission of the same sample is being measured.

2.2 Redeposition Studies.

A physical redeposition study using a UC cathode and a Ni anode has been initiated. The cooling of the anode by compressed air was

* Based on the theoretical density of the nominal composition.

** About 84% density before the vaporization runs, based on the theoretical density of the nominal composition.

found to be inadequate for long-term runs. This is being changed to water cooling through a thermal barrier.

2.3 Thermionic Emission Microscopy.

Preliminary examinations have been made on the emission pattern from the polished surface of a hyperstoichiometric UC sample.

2.4 High-Temperature Mechanical Properties.

The sample testing fixtures were incorporated into the furnace and tested at 1800°C . Minor mechanical difficulties encountered have been solved. Machining techniques for putting flats on cylindrical carbon pieces have been developed for preparing a large number of samples for the test.

3. Irradiation Studies.

3.1 Unclad Carbide Capsule.

The irradiation of the unclad carbide capsule is proceeding satisfactorily in GETR.

3.2 Clad Capsule.

The carbide fuel bodies have been prepared. Cladding of these fuels with vapor-deposited tungsten and machined 98 wt-% W - 2 wt-% Mo alloy is in progress.

4. Studies of New Cathode Materials.

4.1 Vacuum Emission and Vaporization.

The vacuum emission of sample D_2-1 (UMoC_2 , stoichiometric) was measured after the study of its vacuum vaporization characteristics.

4.2 Diffusion.

The compatibility between 98 wt-% W - 2 wt-% Mo alloy and UC-ZrC (90 UC - 10 ZrC and 30 UC - 70 ZrC) was studied (Run D₂-4) at 1800°C for a period of 24 hr. The removal of quenched-in uranium in metal-rich UC and 90 UC - 10 ZrC samples by thermal treatments was investigated.

4.3 Diffusion-Emission.

Sample D₃-1 (vapor-deposited tungsten-clad UC containing 5.02 wt-% carbon, 838 hr at 1800°C) has been set up for microprobe analysis. Two hypostoichiometric UC wafers (4.70 wt-% C) are being coated with tungsten for diffusion-emission studies.

5. Fabrication Development.

Main efforts have been devoted to the fabrication of the samples for the clad irradiation capsule.

I. EXPERIMENTAL PROGRAM.

This monthly progress report covers the work accomplished during the period March 1 to March 31, 1963 under Contract NAS 3-2532. The purpose of this contract is to continue the efforts made under Contract NAS 5-1253⁽¹⁾ and NAS 3-2310⁽²⁾ for establishing the feasibility of the UC-ZrC and the W-clad UC thermionic cathode system and to develop new cathode materials. The subjects to be studied are: (1) long-term (~1000 hr) tests of the vaporization and emission stability of the UC-ZrC and the W-clad UC systems in the presence of cesium vapor; (2) studies of the controlling factors of the vaporization, emission, and mechanical properties of the UC-ZrC systems; (3) studies of the irradiation properties of the UC-ZrC and the W-clad UC systems; (4) studies of the vaporization, emission, and diffusion properties of new cathode materials; and (5) development of fabrication techniques for controlling the structures and

compositions of samples used in these studies.

1. Long-term Tests.

1.1 Rate of Vaporization of UC-ZrC in Cesium Environment.

Previously the presence of cesium vapor at a pressure of 1.8 torr was found to reduce the rate of vaporization of a 90 UC - 10 ZrC sample (A_1 -1a, 93.4% density) at 2093°K by a factor of 6 during a 100-hr run⁽³⁾. To check this finding, another 100-hr run was made under similar conditions at 2083°K with a different 90 UC - 10 ZrC sample (B_1 -3, 95.5% density) whose vacuum vaporization characteristics have been studied during the first quarter of this contract⁽⁴⁾. Prior to the run in cesium, the rate of vaporization of B_1 -3 in vacuum was measured again at 2083°K and found to agree with the previous result. The data obtained in the presence of cesium, however, indicated that the reduction in vaporization loss was only a factor of 1.4 instead of 6. After the cesium run, the vacuum vaporization rate of B_1 -3 was remeasured at 2073°K . Strangely, the rate was five times higher than that for B_1 -3 before the cesium run. The reason for this increase in vaporization rate is not clear at this moment. Studies are being continued on the vacuum vaporization of B_1 -3 to determine whether this increase in rate of vaporization is permanent. Irrespective of what the factor of reduction is, the observed rate of vaporization of B_1 -3 in cesium at 1.8 torr pressure and 2083°K is $9.44 \times 10^{-9} \text{ gm/cm}^2/\text{sec}$, as compared to $7.37 \times 10^{-9} \text{ gm/cm}^2/\text{sec}$ for that of A_1 -1a at 2093°K , both of which are higher than the goal of $10^{-9} \text{ gm/cm}^2/\text{sec}$. Preparation is underway for another 100-hr run with a new 90 UC - 10 ZrC sample at 1.8 torr cesium pressure and 2083°K . Two cells similar to the redeposition cell (Fig. 13 and Fig. 14 of the second quarterly report⁽³⁾) have been fabricated for the 1000-hr runs.

1.2 Emission Stability of UC-ZrC and W-clad UC in Cesium Vapor.

Attempts were made to regenerate the 90 UC - 10 ZrC (A_3 -1) sample in the first life-test cell by thermal treatment after freezing

out the cesium. The check of the emission of the treated emitter was unsuccessful, indicating that leakage probably had developed in some cell components. The test was therefore terminated after about 500 hr and the cell cut open. It was found that leakage had occurred both in the tantalum partition between the cell and the electron-gun compartment and in the tantalum sheath of the thermocouple used for monitoring the emitter temperature. The cause of these leaks was traced to a faulty metal gasket used to seal the joint between the electron-gun compartment and the vacuum system. The gasket developed a small leak which led to the embrittlement of the tantalum partition and the tantalum sheath of the thermocouple.

The second life-test cell is being assembled. A UC wafer clad with vapor-deposited tungsten will be used as the emitter..

2. Studies of the High-Temperature Properties of UC-ZrC.

2.1 Effect of Porosity on the Vaporization and Electron Emission Properties of UC-ZrC in Vacuum.

Measurements have been made on the rate of vaporization of a low-density* cold-pressed and sintered nominal 30 UC - 70 ZrC sample (B_1-9). The results are shown in Table 1 and plotted in Fig. 1 together with those for sample B_1-7 (27.4 UC - 72.6 ZrC, 90% density, open pore = 33.5% of total void) for comparison. It is interesting to note that not only the rate of vaporization of B_1-9 is higher than that of B_1-7 , but also the slope of the plot for B_1-9 is different from that for B_1-7 . Currently the vacuum emission of B_1-9 is being measured. The final composition and pore structures of B_1-9 will be determined after the completion of the emission measurements.

2.2 Redeposition Studies.

A physical redeposition run has been made between a UC cathode at 1730°C and a Ni anode at 780°C in the all-metal cell shown in Fig. 13 and Fig. 14 of the second quarterly report⁽³⁾. The anode

temperature was maintained by adjusting the rate of flow of the compressed
*About 84% density before the vaporization runs, based on the theoretical density of the nominal composition.

air impinging against the back of the anode. The cell operated satisfactorily for a period of 72 hr before a leak developed at the point where the Ni-sheathed Chromel-Alumel thermocouple was welded to the center of the anode surface and where the compressed air coolant impinged on. It is felt that air cooling is probably not suitable for a Ni anode operating at high temperatures. An arrangement for cooling the anode with water through a metal thermal barrier has been constructed. This is being tested at this moment in the same cell containing a UC cathode and a Ni anode.

2.3 Thermionic Emission Microscopy.

A hyperstoichiometric UC sample was polished and examined in the microscope at 1700°K . Heterogeneity of the emitting surface was demonstrated and fine pearlite-like structures were observed in some of the crystal grains. The emission pattern changed gradually under continuous heating and applying the accelerating potential. In a few hours, the surface of the sample seemed to become quite porous. Correlation of these observations with metallographic examinations is being pursued.

2.4 High-Temperature Mechanical Properties.

The sample testing fixtures have been incorporated into the furnace and heated to 1800°C for "shakedown" evaluation of the equipment. Minor mechanical difficulties were observed in the loosening of the bottom testing fixture and swelling of the sample loading tube in its guide. In addition, the bottom of the furnace was found to be uneven so that the stability of the whole furnace may be affected during the testing. Necessary corrections and refitting were therefore made and all these difficulties have been overcome.

Machining techniques for putting flats on the 1/4-in.-diameter and 1-1/4-in.-long test bars have been developed using electrodischarge machining. Six bars can be machined in one operation. Tests on 10 UC - 90 ZrC bars will be initiated shortly.

3. Irradiation Studies.

3.1 Unclad Carbide Capsule.

The irradiation of the unclad carbide capsule GA2-306-1F1 started in GETR on March 4, 1963. All the thermocouples operated satisfactorily. The temperatures recorded by each of the four W- (74 W-26 Re) thermocouples during March, 1963, are plotted in Fig. 2 (a), (b), (c) and (d). The RAFT facility was not in use until March 8, 1963, because of the need of some additional thermal stress calculations by GETR before the temperatures of the outer Inconel can could be raised to above 1000°F. Although it is too premature to speculate on the conditions of the samples in each pin from the thermocouple data shown in Fig. 2, the information does indicate that:

(1) All the thermocouple readings declined near the end of the reactor cycle, probably due to the change of the neutron flux density and distribution in the reactor.

(2) The low-density pin containing No. 10 thermocouple stayed in the temperature range of 1700 to 1800°C for most of the cycle.

(3) The temperature of the high-density pin, as shown by the readings of thermocouple No. 9, decreased steadily from March 8 to below 1300°C near the end of the cycle, indicating an increasing loss of heat (probably due to fuel swelling) from the high-density carbide samples.

(4) The two thermocouples (No. 7 and No. 8) in the medium-density pin, for some unknown reasons, do not register similarly. The readings of both dropped to about 1450°C near the end of the cycle, probably due to the change of the neutron flux conditions in the reactor.*

The second cycle will commence during the first week of April and will end by about the first week of May, 1963.

* The temperature drop is probably not due to fuel swelling, since when GETR restarted, the temperatures of thermocouples No. 7 and No. 8 went back to 1700°C and 1650°C, respectively. Thermocouple No. 9, however, registered only 1350°C.

3.2 Clad Capsule.

3.2.1 Vapor-deposited tungsten-clad samples.

All the carbide fuel bodies have been prepared. Cladding of these fuel pieces by vapor-deposited tungsten according to the configuration shown in Fig. 30 (b) of the second quarterly report⁽³⁾ ran into some difficulties. It was discovered that H_2F_2 which is the gaseous product of the coating process using $WF_6 + H_2$, became trapped inside the void maintained by the tungsten preform and severe corrosion of the carbide resulted. This difficulty was overcome by first sealing the carbide with a thin layer (0.003 in.) of tungsten under conditions where the contact between the carbide and the H_2F_2 formed was minimized. During the buildup of the thickness of the coating by the normal procedures, this thin seal-coating prevented the H_2F_2 trapped inside the void from reacting with the carbide. Since the seal-coating is very thin, it is felt that it should not stop the fission gases from leaving the fuel and going into the void once the fission gas pressure is built up in the fuel. Because of the addition of this seal-coating, the tungsten baffles used for limiting fuel redistribution in the clad can during the irradiation are eliminated.

The coating gases consist of a mixture of $WF_6 + H_2 + He$ at a total pressure of about 1/2 atm., which has been freed from oxygen and moisture contaminations. During the seal-coating period, the velocity of the gas flow is maintained high in order to sweep away as far as possible the corrosive reaction product H_2F_2 formed, and the temperature of the sample is maintained low (350 to 400°C) so that the rate of H_2F_2 formation is low. Because of the low temperature used, the rate of coating is consequently also low. The seal-coated sample is then placed inside the tungsten preforms and encapsulated at about 700°C. After the coated sample is cooled to room temperature, a small hole is drilled into one of its flat faces, and the sample is outgassed at 1800°C. The hole is then plugged with a small tungsten pin and sealed with Pt braze.

Several seal-coated specimens have been examined; no attack of the carbide was observed. Two seal-coated specimens were placed in preforms, encapsulated and outgassed as described above. After sectioning, the carbide pieces were found to be in very good shape. These results establish the soundness of the procedures mentioned above for the cladding of carbide fuels with tungsten by the thermochemical technique.

Coating of the UC, 90 UC - 10 ZrC, 30 UC - 70 ZrC samples is being carried out at San Fernando Laboratory. The coating of UO_2 spherical particles by vapor-deposited tungsten has also been attempted at San Fernando Laboratory. Results obtained to date indicate that a certain amount of developmental work is needed before a satisfactory procedure can be established. To meet the irradiation schedule, both the W- UO_2 and the W-UC cermet will be fabricated from a blended mixture of tungsten powder and the fuel particles, as described in the second quarterly report⁽³⁾.

3.2.2 98 wt-% W - 2 wt-% Mo alloy clad samples.

The samples are being fabricated according to the configuration of Fig. 33 (b) in the second quarterly report⁽³⁾. All the alloy cups and caps have been machined. Compatibility studies as described in the second quarterly report⁽³⁾ and in Section 4.2 of this report, indicate that UC and 90 UC - 10 ZrC interact with this alloy at 1800°C and that a very slight amount of interaction has also been observed between this alloy and 30 UC - 70 ZrC. On the basis of these observations, it has been decided that the fuels used in the alloy clad irradiation samples will be limited to 30 UC - 70 ZrC, W-UC cermet, and W- UO_2 cermet. Furthermore, the inside surface of each alloy can will be coated with 0.003-in.-thick vapor-deposited tungsten to minimize the chance of clad-fuel interaction. Since the fuel pieces will not be seal-coated with vapor-deposited tungsten, tungsten baffles will be used to limit the redistribution of fuels in the clad can during the irradiation. The 30 UC - 70 ZrC fuel pieces have been fabricated. The cermet fuels, as stated in Section 3.2.1, will

be fabricated from a blended mixture of tungsten powder and the fuel particles. Tungsten coating of the inside surface of the alloy cups is being carried out at San Fernando Laboratory. After the fuel pieces are loaded into the alloy cups, the caps will be diffusion-bonded to the fueled cups by using 74 W - 26 Re alloy as an intermediate layer, as stated in the second quarterly report⁽³⁾.

4. Studies of New Cathode Materials.

4.1 Vacuum Emission and Vaporization.

The vacuum emission of the hot-pressed stoichiometric UMoC_2 sample D_1-2 (88.9% density) has been measured after the completion of its vacuum vaporization studies. The results are shown in Fig. 3. Included in the same figure for comparison are the vacuum emission data of the hot-pressed carbon-rich UMoC_2 sample D_1-1 ($\text{U}_{0.24}\text{Mo}_{0.22}\text{C}_{0.54}$) and the vacuum emission data for the hot pressed 30 UC - 70 ZrC (90% density) sample B_1-7 . These results fall closely to one another, supporting the theory that the emission is primarily due to the presence of uranium in the sample. UC-NbC and UC-TaC containing 90 mol-% UC and 30 mol-% UC have been prepared by cold-pressing and sintering. Studies of the vacuum vaporization rates of the UC-NbC samples will be initiated as soon as the carbide pieces are cavitroned to the desired size.

4.2 Diffusion.

4.2.1 Studies of removal of quenched-in uranium from metal-rich UC and UC-ZrC by thermal treatments.

In the second quarterly report⁽³⁾, two compatibility runs at 1800°C for 24 hr were carried out to study the suitability of 85 wt-% W - 15 wt-% Mo and 98 wt-% W - 2 wt-% Mo alloys as candidates for the cladding of

uranium carbide reactor fuels.

Briefly, the results may be summarized as follows:

(1) UC, in contact with both 98 wt-% W - 2 wt-% Mo and 85 wt-% W - 15 wt-% Mo, developed an extensive grain boundary liquid phase associated with radical grain growth.

(2) Both 98 wt-% W - 2 wt-% Mo and 85 wt-% W - 15 wt-% Mo alloys were attacked by reaction with the uranium carbide and were penetrated throughout by a liquid phase thought to be a uranium-molybdenum alloy. This liquid phase was seen at a large percentage of the triple grain boundary intersections.

(3) UC in contact with pure tungsten showed liquid-phase formation. The liquid did not penetrate the tungsten.

Several hypotheses were made to explain the formation of a liquid phase in the uranium carbide. These were: (1) The single-phase structure of the metal-rich carbide used was due to a quenched-in high-temperature solubility for uranium metal which easily migrated to the 98 wt-% W - 2 wt-% Mo or 85 wt-% W - 15 wt-% Mo alloy and alloyed with Mo.

(2) Nitrogen and oxygen were present in the uranium carbide in sufficient quantities to hold uranium as UN and UO in solution with UC to give a single-phase structure. It was then gettered by the test alloys to precipitate liquid uranium. (3) Carbon, because of an activity gradient, migrated from the carbide to the test alloys, leaving the carbide so metal-rich as to precipitate uranium.

During this reporting period, procedures were established and completed to check the first hypothesis.

Thermal treatment of uranium carbide.

A sample of the metal-rich uranium carbide (4.76 - 4.80% C) used for the compatibility runs referred to above was supported on ZrC and heated to 1800°C for 2 hr, slowly cooled to 1100°C over a period of 2 hr, and then furnace-cooled to room temperature. The treatment was designed to remove a quenched-in structure and allow uranium to precipitate.

Metallographic examination of the heat-treated specimen revealed that a liquid phase was present at the edges of the specimen. Little grain growth had occurred. These results seemed to partially substantiate the hypothesis of a quenched-in high-temperature solubility for uranium.

Thermal treatment of 90 UC - 10 ZrC.

Several samples of 90 UC - 10 ZrC were obtained to evaluate the compatibility of this fuel material with 98 wt-% W - 2 wt-% Mo. The material was free from uranium inclusions and metal-rich (4.74 - 4.8 wt-% C, stoichiometric sample C = 5.01 wt-%).

In order to fully characterize the material prior to the compatibility run, the following tests were performed:

(1) A sample of the above chemistry, supported on clean ZrC, was heated to 1500°C for 1/2 hr, then cooled to 1270°C to 1290°C and held for 8 hr to precipitate any quenched-in metal. Metallographic examinations showed only trace amounts of precipitated metal at a few grain boundaries. Chemical analyses showed that the heat-treated material had essentially the same carbon content (4.72 to 4.76 wt-% C) as before the heat treatment. This lack of change in the carbon content indicates that the material was not benefited by the evaporation of uranium from the sample during this treatment.

(2) The 90 UC - 10 ZrC used for the compatibility test with 98 wt-% W - 2 wt-% Mo was subjected to a heat treatment designed to remove any free uranium from the structure by evaporation. The material was heated to 1800°C in a cold wall vacuum chamber for 1 hr and then cooled for 1 hr at each of the following temperatures: 1700°C, 1470°C, 1370°C, and 1270°C. Analyses for carbon, nitrogen, and oxygen are being obtained for this material.

4.2.2 New Compatibility Tests at 1800°C (Run D₂-4).

A further series of specimens was employed to determine if the incompatibility of 98 wt-% W - 2 wt-% Mo alloy with UC would

extend to uranium carbide fuel materials stabilized with ZrC. To accomplish this evaluation, 90 UC - 10 ZrC and 30 UC - 70 ZrC were placed in contact with 98 wt-% W - 2 wt-% Mo alloy at 1800°C for 24 hr in the following array:

W	W	30 70	W	W- 2 Mo	30 70	W- 2 Mo	W	W- 2 Mo	90 10	W- 2 Mo	W
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A 30 UC - 70 ZrC versus W sample was included as a comparison standard; this combination is considered to be compatible for the test conditions. The results obtained are described as follows.

90 UC - 10 ZrC versus 98 wt-% W - 2 wt-% Mo.

(1) An extensive liquid phase was formed surrounding the 90 UC - 10 ZrC grains in amounts much greater than could be generated by precipitation heat treatment of the 90 UC - 10 ZrC not in contact with the alloy. Radical grain growth occurred in the 90 UC - 10 ZrC. Visual estimation of the amount of liquid present showed less than the amount present with pure UC in contact with 98 wt-% W - 2 wt-% Mo for similar test conditions.

(2) A liquid phase, thought to be a U-Mo alloy, was found throughout the 98 wt-% W - 2 wt-% Mo alloy at triple grain boundary intersections. This liquid phase extended to and completely filled the W to 98 wt-% W - 2 wt-% Mo interface.

30 UC - 70 ZrC versus 98 wt-% W - 2 wt-% Mo.

(1) The 30 UC - 70 ZrC showed essentially a single-phase structure. No large amounts of liquid phase were observed.

(2) The 98 wt-% W - 2 wt-% Mo revealed very small amounts of liquid phase at triple grain boundary intersections throughout its volume.

30 UC - 70 ZrC versus W.

No liquid-phase formation was observed either in the carbide or in the tungsten.

Thus, 98 wt-% W - 2 wt-% Mo alloy does not appear to be as attractive a clad material as pure tungsten for uranium carbide fuels, even when

diluted with as much as 70 mol-% ZrC, for reasons of a possible degradation of strength due to the presence of a grain boundary liquid phase and because fissioning would occur in this liquid phase if indeed it does contain uranium. The incompatibility does not seem to be related to a quenched-in high-temperature solubility for uranium metal by either pure UC or 90 UC-10 ZrC.

4.3 Diffusion-Emission.

Sample D₃-1, a hyperstoichiometric (5.02 wt-% C) UC wafer clad with vapor-deposited tungsten, which has been heated for 838 hr at 1800°C, has been set up for microprobe analysis of any uranium penetration into the tungsten. Two hypostoichiometric (4.70 wt-% C) UC wafers are being coated with vapor-deposited tungsten at San Fernando Laboratory for continuing the diffusion-emission studies of the W versus UC system.

FUTURE PLANS

1. Long-term Tests.

1.1 Rate of Vaporization of UC-ZrC in Cesium Vapor.

90 UC - 10 ZrC and 30 UC - 70 ZrC samples of 85 to 90% theoretical densities will be prepared by cold-pressing and sintering for the 1000-hr runs in the two all-metal cells built for such a purpose. Studies of the vacuum vaporization rate of sample A₁-1a will be continued in order to find out whether the increase in its vacuum vaporization rate by heating in cesium is permanent. Another 100-hr run will be made at 1.8 torr cesium pressure and 2083°K with a new 90 UC - 10 ZrC sample.

1.2 Emission Stability of UC-ZrC and W-clad UC in Cesium Vapor.

The second life-test cell containing a vapor-deposited tungsten-clad UC cathode will be assembled and operated. Parts for the third life-test cell will be gathered for its assembling.

2. Studies of High-temperature Properties of UC-ZrC.

2.1 Effect of Porosity on the Rate of Vaporization and Electron Emission of UC-ZrC in Vacuum.

Measurements will be made on the vacuum emission of the low-density 30 UC - 70 ZrC sample B₁-9, the vacuum vaporization rate of which has been described in this report. The final composition and the pore structures of B₁-9 will be determined. Studies will be initiated on the vacuum vaporization rate and vacuum emission of another low-density 30 UC-70 ZrC sample B₁-10 prepared by cold-pressing and sintering.

2.2 Redeposition Studies.

Physical redeposition studies will be carried out between a UC cathode at 1800°C and a Ni anode at various temperatures between 300 to 900°C.

2.3 Thermionic Emission Microscopy.

Efforts will be made to correlate the emission pattern and the microstructures of UC. Studies will be initiated on the poisoning and regeneration of a UC emitter.

2.4 High-temperature Mechanical Properties.

Measurements will be started on the modulus of rupture of the 10 UC - 90 ZrC samples.

3. Irradiation Studies.

3.1 Unclad Carbide Capsule.

The conditions of operation of the capsule in GETR will be followed during its second irradiation cycle. Plans for the hot-cell examination of this capsule will be laid down, in anticipating the discharge of this capsule by the first week of May, 1963.

3.2 Clad Capsule.

The samples of the clad capsule will be fabricated and the capsule components gathered for its assembling by the first week of May, 1963.

4. Studies of New Cathode Materials.

4.1 Vacuum Emission and Vaporization.

The vacuum rate of vaporization of the UC-NbC sample will be initiated.

4.2 Diffusion.

Samples for diffusion runs D_2-1 (Re versus UC and 75 W - 25 Re versus UC), D_2-2 (85 wt-% W - 15 wt-% Mo versus UC), D_2-3 (98 wt-% W - 2 wt-% Mo versus UC) and D_2-4 (98 wt-% W - 2 wt-% Mo versus 90 UC - 10 ZrC) will be sent for microprobe studies for establishing concentration gradients and compositions of new phases. The W-Mo alloys (2 and 15 wt-% Mo) used in the diffusion runs D_2-2 , D_2-3 and D_2-4 will also be studied for any segregation of Mo at their grain boundaries.

4.3 Diffusion-Emission.

Sample D_3-1 (vapor-deposited tungsten-clad hyperstoichiometric UC) will be subject to microprobe studies for any uranium diffused into the tungsten clad during its 838 hr at 1800°C . Samples D_3-2 and D_3-3 (vapor-deposited tungsten-clad hypostoichiometric UC) will be prepared and the studies initiated at 1800°C .

5. Fabrication Development.

The main efforts will be in the fabrication of the samples of the clad irradiation capsule.

REFERENCES

1. Final Report for Contract NAS 5-1253, GA-3523.
2. Final Report for Contract NAS 3-2310, GA-3642.
3. Second Quarterly Report for Contract NAS 3-2532, GA-4173.
4. First Quarterly Report for Contract NAS 3-2532, GA-3866.

Table 1

VAPOR LOSS INVESTIGATION OF 30 mol-% UC - 70 mol-% ZrC SAMPLE NO. B₁-9

Run Number	Temp. (°K)	Exposure Time (sec x 10 ⁻⁴)	Rate of Weight Loss (mg/cm ² /sec) x (10 ⁻⁵)	Counting Rate Alpha Counts/min/cm ²		UC Surface Concentration Based on Counting Rate (mol-%)	Density ₃ gms/cm
				Side 1	Side 2		
Original	-	-	-	964	973	15.5	7.56
Degas	2193	9.54	9.99	536	544	3.5	7.58
1	2273	2.52	6.45	493	508	3.0	7.54
2	2163	4.38	2.55	446	466	7.5	7.64
3	2245	2.88	4.43	454	464	7.5	7.73
4	2128	2.88	1.85	450	467	7.5	7.72
5	2073	3.42	1.34	453	447	7.0	7.71

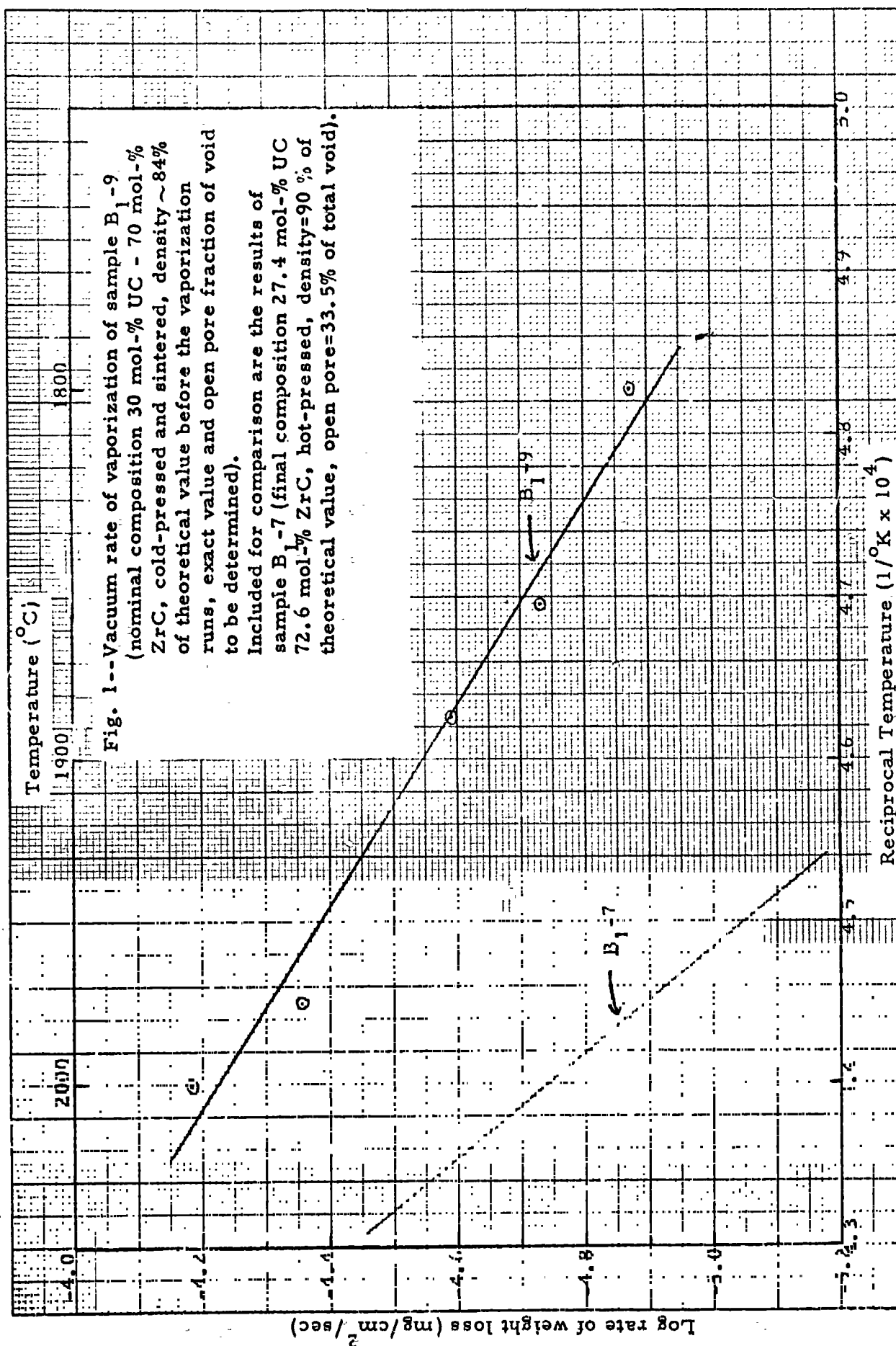
Note:

Original composition based on chemical analysis: U_{0.151} Zr_{0.367} C_{0.482}

X-ray diffraction analysis performed prior to vapor-loss determinations indicated a composition of about 30 mol-% UC - 70 mol-% ZrC, single phase.

Based on 30 mol-% UC - 70 mol-% ZrC the specimen was 83.5% dense prior to vapor-loss investigations.

Final composition will be reported after the sample is analyzed.



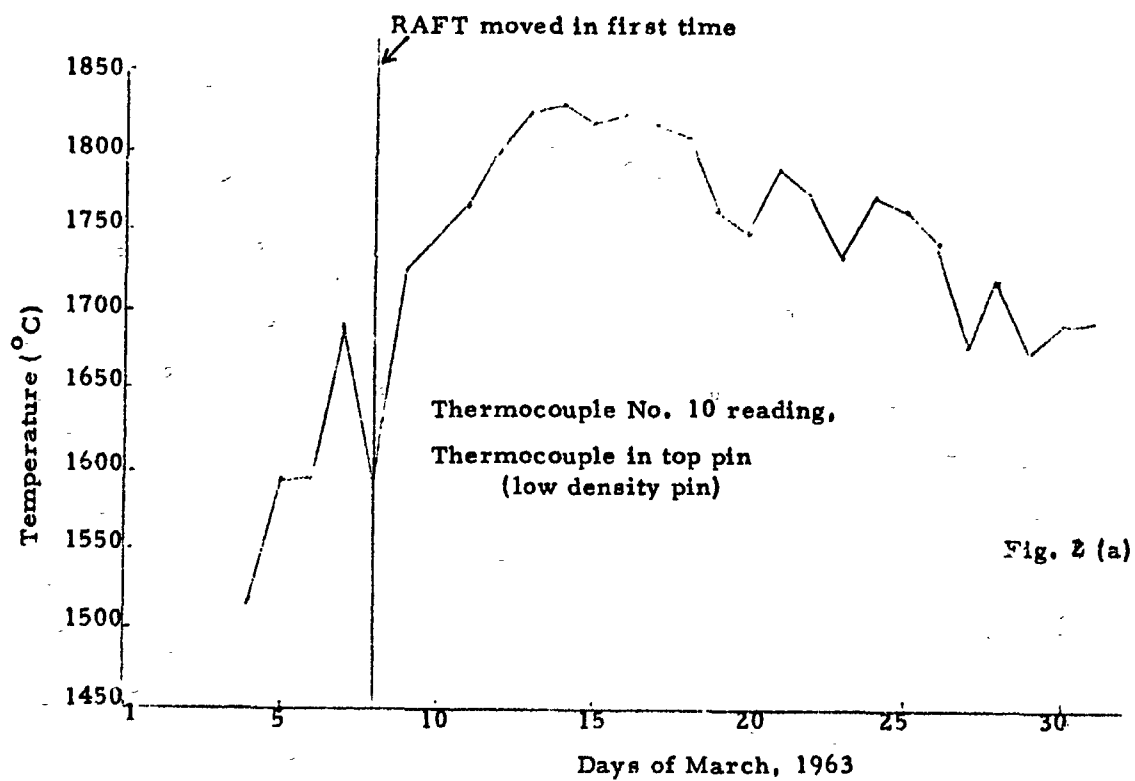


Fig. 2--Temperatures of pins in the unclad carbide capsule.

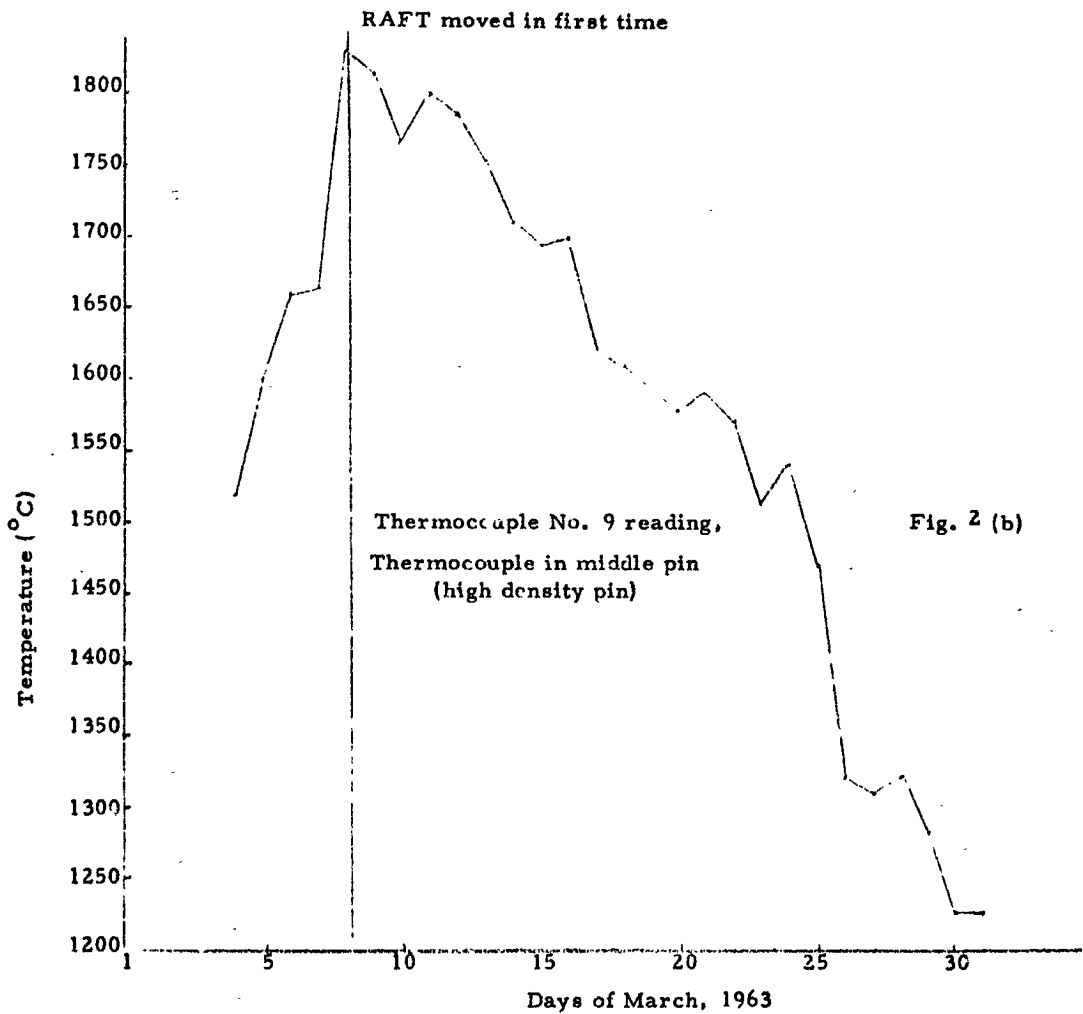
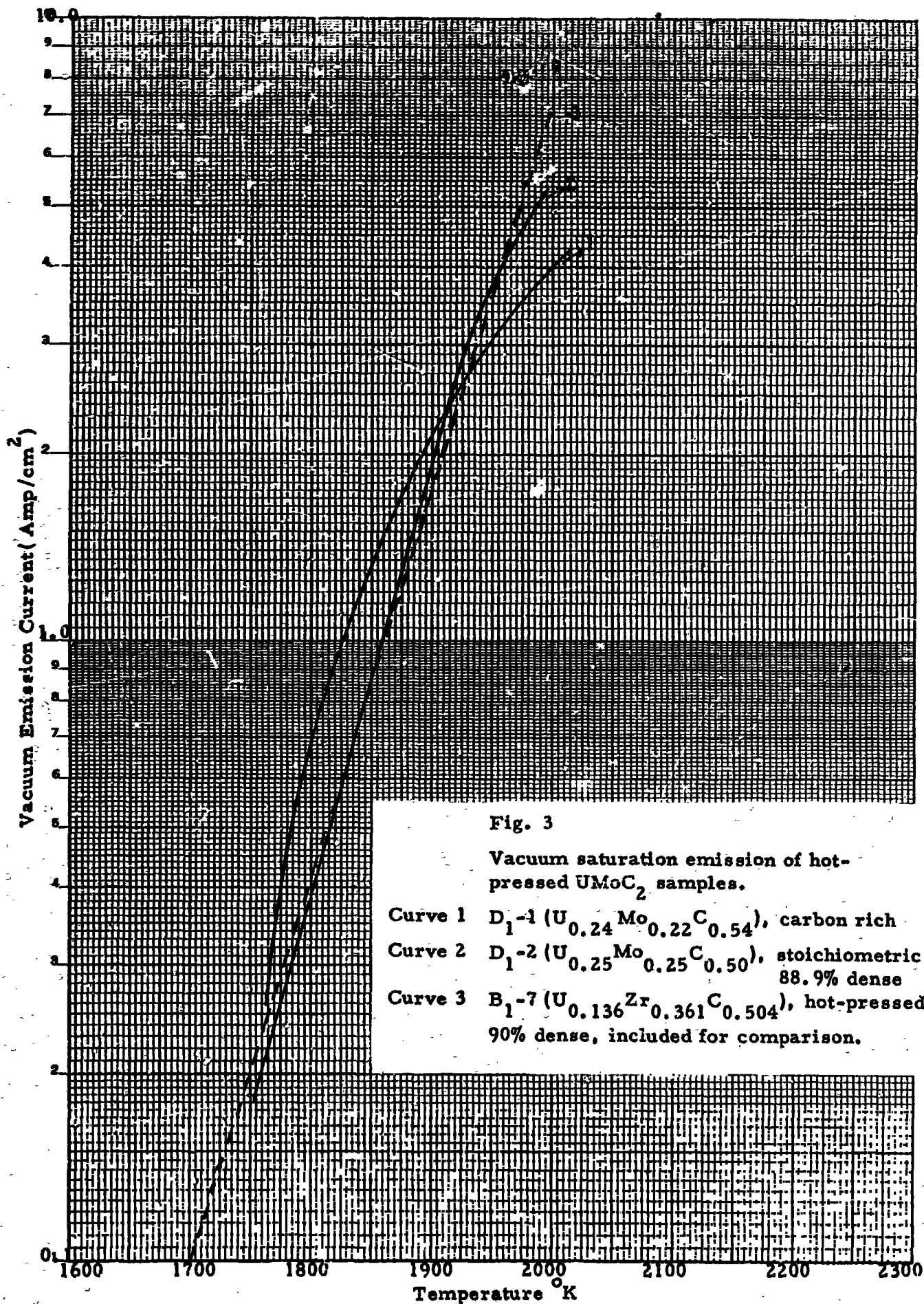


Fig. 2(cont.)--Temperatures of pins in the unclad carbide capsule.



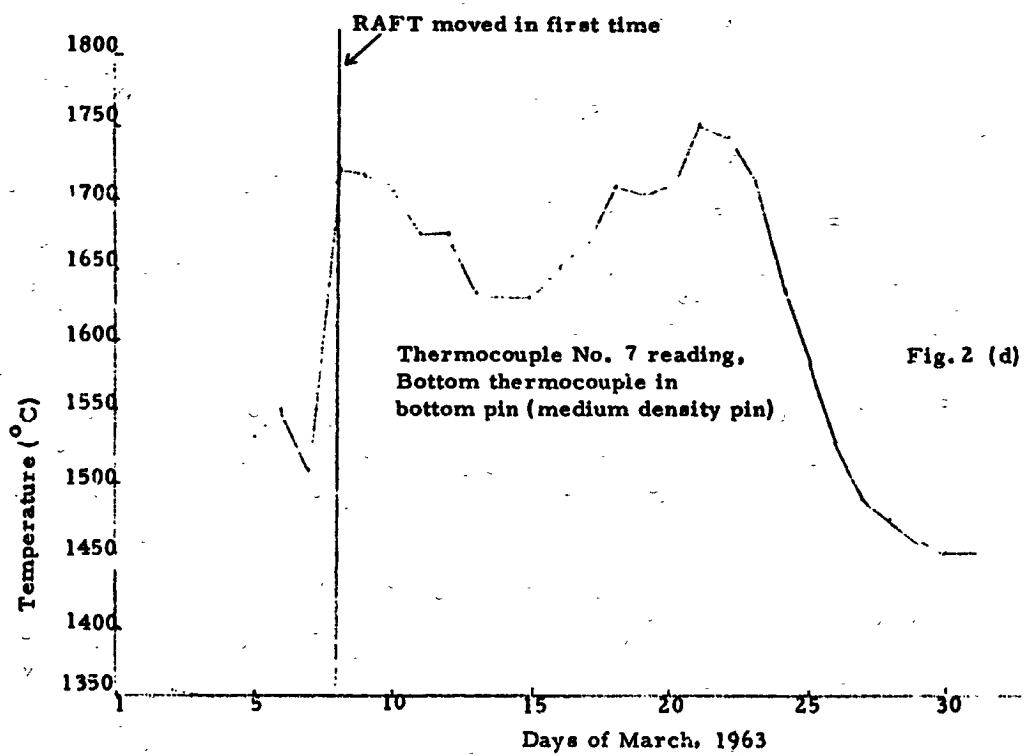
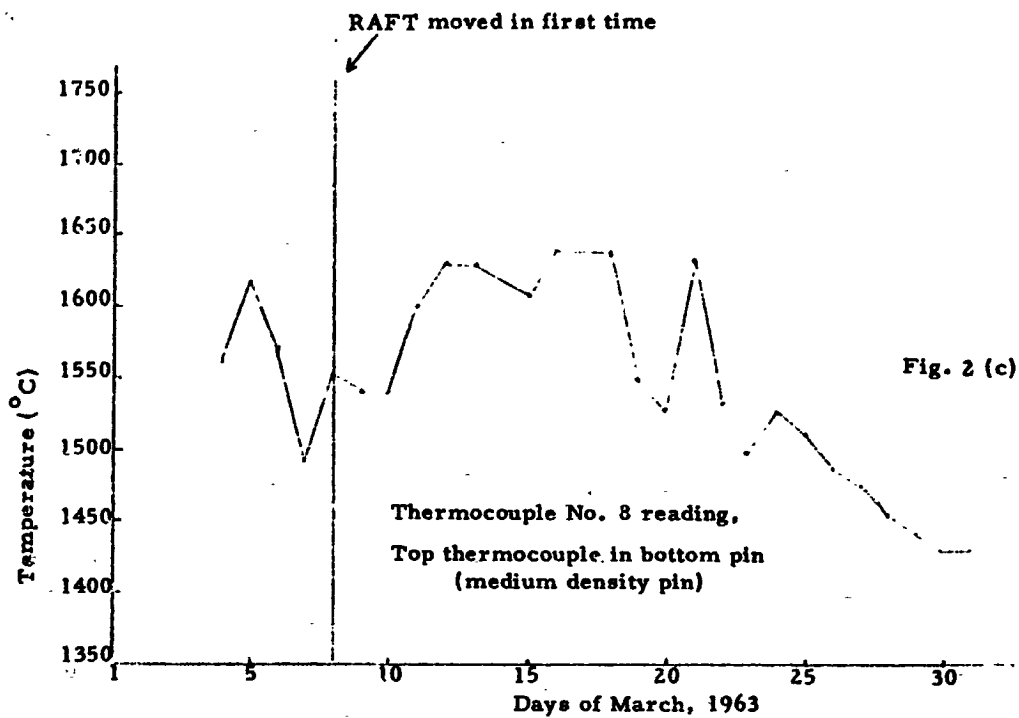


Fig. 2(cont.)--Temperatures of pins in the unclad carbide capsule.